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Respirable Size-Selective Sampler for End-of-Shift Quartz Measurement: Development and Performance

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Abstract

Aims of this study were to develop a respirable size-selective sampler for direct-on-filter (DoF) quartz measurement at the end-of-shift (EoS) using a portable Fourier transform infrared (FTIR) spectrometer and to determine its size-selective sampling performance. A new miniaturized sampler has been designed to have an effective particle deposition diameter close to the portable FTIR beam diameter (6-mm). The new sampler (named the EoS cyclone) was constructed using a 3 dimensional printer. The sampling efficiency of the EoS cyclone was determined using polydisperse glass sphere particles and a time-of-flight direct reading instrument. Respirable dust mass concentration and quartz absorbance levels of samples collected with the EoS cyclone were compared to those collected with the 10-mm nylon cyclone. The EoS cyclone operated at a flow rate of 1.2 l min^{-1} showed minimum bias compared to the international standard respirable convention. The use of the EoS cyclone induced respirable dust mass concentration results similar but significantly larger (5%) than those obtained from samples collected with 10-mm nylon cyclones. The sensitivity of the DoF-FTIR analysis in estimating quartz was found increased more than 10 times when the samples were collected with the EoS cyclone. The average particle deposition diameter was 8.8 mm in sixty samples. The newly developed user friendly EoS cyclone may provide a better sampling strategy in quartz exposure assessment with faster feedback.

Keywords

End-of-Shift; cyclone; quartz; respirable size-selective sampler; silica

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DISCLAIMER

The findings and conclusions in this report are those of the author(s) and do not necessarily represent the views of the National Institute for Occupational Safety and Health.

INTRODUCTION

The National Institute for Occupational Safety and Health (NIOSH) is conducting research to eliminate respiratory diseases in mine workers by reducing exposure to airborne contaminants. This research includes developing field-ready instrumentation capable of an end-of-shift (EoS) measurement for respirable crystalline silica contained in the dust present in the mine atmosphere.[1] Silica exposure levels of mine workers can then be determined at EoS or during-shift (DS) whereas current methods require a longer off-site analysis. Field-portable spectrometers including a Fourier transform infrared (FTIR) spectrometer, a variable filter array (VFA) spectrometer, and a Laser-Induced Breakdown spectrometer (LIBS) have been investigated for EoS analysis.[2, 3] It was determined that the field-portable FTIR spectrometer gave relatively accurate analyses of filter samples using a direct-on-filter (DoF) sample collection method. Portable FTIR spectrometers are small, battery-operated, relatively easy to use, and cost less than laboratory instruments.[1] Quartz measurements in respirable coal mine dusts using the DoF method and standard indirect method (ashed sample to remove organic materials and redeposited on a filter) have been compared. Their correlation coefficients were found to be close to unity[4], suggesting that concerns about interference of IR absorption by coal dust might be minimal in the DoF method. The Institut National de Recherche et de Sécurité (INRS, France) reported that underestimation (average of 13%) of the indirect IR method compared to average value of DoF methods with XRD and IR was found due to either elimination of organic compounds or by the transformation of mineral matrices during calcination of the dust rather than loss of material during preparation.[5] Field-portable FTIR instruments are less sensitive than laboratory units and are also incapable of analyzing the entire amount of collected samples on the filter due to differences in area between the particle deposition by respirable size-selective samplers and the FTIR beam. For example, a 10-mm nylon cyclone standard respirable size-selective sampler[6], provides a 34-mm diameter particle deposition area on-filter while the beam diameter of a commercially available portable FTIR spectrometer is about 6 mm. Deposition profiles of respirable coal dust, with sample-to-sample variation, changed slightly as filter loading increased when samples were collected with 10-mm nylon cyclones.[7] Different deposition profiles of coal dust were also found on filters from different sampling cassettes (2-piece, 3-piece, MSA cassette) collected with 10-mm nylon cyclone.[7] The deposition profiles generated by various low-flow and high-flow rate samplers were also investigated with respirable metal mine dust.[8] Most of the samplers employ 37-mm filters, and filters are usually removed from filter cassettes for EoS quartz measurements. If quartz masses were increased in the FTIR beam path by decreasing the particle deposition area, larger FTIR absorbance might be obtainable, consequently reducing relative standard deviations in quartz measurements using the portable FTIR.

The objectives of the present study were 1) to develop a user-friendly respirable size-selective sampler whose particle deposit diameter is close to that of a field-portable FTIR beam to allow EoS quartz measurement without removing the filter from the sampling cassette and 2) to determine the size-selective sampling performance of the new sampler.

METHODS

Production of cyclone using a three dimensional printer

The baseline for our cyclone body and vortex finder designs was adapted from a multi-inlet cyclone (GS3 cyclone, SKC, Inc. Eighty Four, PA, USA), which is considered an improvement over the design of the 10-mm nylon cyclone (single inlet).[9, 10] The three inlet design was used to minimize the effect of inlet orientation on aspiration efficiency. The internal dimensions for GS3 cyclone were scaled to two-thirds.

Several revisions to any prototype would likely be required to reach the desired performance and to obtain a proper fit between parts. This assumption proved to be correct, despite baselining many internal dimensions from previous work.[9] Cost and turn-around time to produce and test several versions would have been prohibitive had conventional manufacturing methods such as molding or wire electrical discharge machining been employed. Instead, three dimensional (3D) printing was utilized. Prototypes were printed at the NIOSH facility in Pittsburgh, PA on a Fortus model 360mc 3-D printer loaded with ABS-M30 material (Stratasys, Inc. Eden Prairie, MN, USA). The model 360mc was setup to print with a 0.005 inch (0.127 mm) layer thickness. Surface roughness was clearly visible and easily detected by touch.

The assembled sampler and an exploded-view drawing are shown in Figure 1. Five parts were constructed on the 3D printer: the cyclone body, vortex finder, filter holder, filter holder ring, and lid. The removable vortex finder allows for easy cleaning of the cyclone body and testing of variant designs. The filter holder, together with the filter element and filter holder ring form the filter assembly. A barb fitting that threads into the lid allows convenient connection of the sampling pump via 3/16 inch inner diameter tubing. Two custom gaskets were constructed using a Mayhew Pro 66002 hollow punch set (Mayhew Steel Products, Inc., Turner Falls, MA, USA).

After sampling, the filter assembly (filter holder, 25-mm filter, and filter holder ring from the Figure 1) can be removed from the cyclone and placed in a custom-made cradle for the field-portable FTIR quartz measurement. The cradle was designed to place the filter at the focal plane of the IR beam in the sample compartment of the portable FTIR. This assures an optimal analysis of the sample on the filter. After the non-destructive analysis in the field-portable FTIR, the filter can be subsequently removed from the filter assembly for further analysis.

Sampling efficiency testing

The size-selective sampling efficiency of the EoS cyclone was investigated as a function of aerodynamic particle size. Sampling efficiencies were determined through experiments with polydisperse glass sphere particles (a mixture of P2011SL and P2015SL, geometric $d_{50}\approx 4$ and 10 μm, respectively, Cospheric, Santa Barbara, CA, USA). Measurements were made using a time-of-flight direct reading instrument (Aerodynamic Particle Sizer (APS), Model 3321, TSI Inc., Shoreview, MN, USA) connected to an aerosol diluter (Model 3302A, 20:1 dilution capillary, TSI Inc.). The test aerosol was generated by a fluidized aerosol generator (Model 3400, TSI Inc.); the dry air feed was provided through a high-efficiency particulate air (HEPA) filtered air supply unit (Model 3074, TSI Inc.). The aerosol was introduced into a calm air chamber (A) of dimensions 28 cm diameter and 68.5 cm length[11, 12] through a Kr-85 aerosol neutralizer (Model 3054A, TSI Inc.) and a baffle allowing the airflow to enter the chamber in a radial direction. A schematic of the test system is shown in Figure 2. As the APS operates with a flow rate of 5 l min^{-1}, HEPA filtered make-up air was provided to adjust the test flow rate at the inlet of the cyclones and reference sample tubing to desired values. The flow rate of the make-up air was controlled by mass flow controllers (MFC; Model GFC-17/37, Aalborg Instruments & Controls, Inc., Orangeburg, NY) connected to house air. The EoS cyclone and a reference sampler (thin-wall tubing, 11 mm inlet diameter) were placed horizontally inside the chamber positioned in the same sampling plane. The flow rate of the reference sampler was the same as the test cyclone and the inlet diameter for the reference sampler was calculated to minimize sampling bias due to settling velocity and inertia of the particles (<20 μm) in calm air conditions.[13, 14] The flow rates before and after sampling were measured with a mass flow calibrator (model 4043, TSI Inc.). Five EoS cyclones were tested. A total of 42 APS measurements were made ((3 reference sampler + 3 EoS cyclones) × 7 repetitions) for each cyclone. Particle number concentration was checked for both tubes without a cyclone attachment (Figure 2) prior to the experiment and the ratio of aerosol concentration was found near unity in multiple measurements. This shows that aspiration efficiencies were nearly the same for both positions and that spatial variability in the testing zone was minimal.

The average measured sampling efficiency of EoS cyclone was compared to the American Conference of Governmental Industrial Hygienists (ACGIH)[15]/Comité Européen de Normalisation (CEN)[16]/International Standards Organization (ISO)[17] respirable convention by calculating the bias.[18–20] Lognormal distribution was assumed and the calculation ranges of mass median aerodynamic diameter (MMAD) and geometric standard deviation (GSD) were 1–30 μm and 1.5–4.0, respectively.

Respirable dust collection

The respirable dust sample collection performance of the EoS cyclones was tested in a calm-air chamber (B; Marple chamber).[7] The chamber, with a hexagonal cross section is 2.44 m high with an inside diameter of 1.19 m. The dust was introduced at the top of the chamber and thoroughly mixed in this region by the energy of an air jet entering at the side of the chamber. From this mixing area, the dust flows downward through a 10-cm thick honeycomb structure where turbulence in the air is reduced, providing a low velocity downward flow through the test section portion of the chamber. The samplers were positioned on a structure around the chamber supported by a rotating table, reducing the effects of any variation in the dust concentration within the chamber. The sampling zone of the chamber, even without rotation, provided spatial uniformity; the relative standard deviation between samples was < 5%.

Three different dusts including a coal dust (pulverized and sieved from Pittsburgh seam coal), a high-purity silica dust (U.S. Silica, Frederick, MD, USA), and a gold mine dust (collected in Alaska gold mine) were aerosolized using a fluidized bed dust generator (3400A, TSI Inc.) through an aerosol neutralizer (3012A, TSI Inc.). Mass median aerodynamic diameter (geometric standard deviation) of the airborne coal dust, high-purity silica dust, and gold mine dust were 5.5 μm (3.2), 3.46 μm (1.73), 5.17 μm (1.72), respectively. The performance of the EoS cyclone was compared to the results obtained by a 10-mm nylon cyclone (Zefon International Inc., Ocala, FL). The 10-mm nylon cyclone is the standard sampler for Occupational Safety and Health Administration (OSHA) method PV2121[21], NIOSH Manual of Analytical Method 0600[6], and Mine Safety and Health Administration (MSHA) methods P-7[22] and P-2[23]. A total of ten cyclones, five 10-mm nylon and five EoS cyclones, were placed in the chamber. Six tests were conducted with each dust: a total of eighteen tests. Two different filter mass loadings were targeted (0.750 mg and 1.5 mg) and each mass loading was obtained for three tests.

The respirable dust mass concentration in the chamber was maintained near 4.5 mg m^{-3} through the entire test. The concentration in the chamber was monitored using a tapered element oscillating microbalance (TEOM) 1400a (Thermo Scientific, Franklin, MA, USA) with a BGI-4CP cyclone inlet operated at 2.2 l min^{-1}.

Flow rates for the 10-mm nylon and EoS cyclones were 1.7 and 1.2 l min^{-1}, respectively, and pre- and post-flow rates were measured using a calibrator (Gilibrator, Gilian Instrument Corp., Wayne, NJ, USA) and a calibration jar (SKC Inc.). For the 10-mm cyclones, samples were collected on 5-μm pore size 37-mm PVC filters (GLA5000, SKC Inc.) placed in conductive cassettes to reduce the wall losses.[24] For the EoS cyclones, samples were collected on 5-μm pore size 25-mm PVC filters (GLA5000, SKC Inc.).

Before and after each test, the PVC filters were equilibrated, neutralized, and pre- and post-weighed in a controlled environment set at 22 ± 0.7°C and 50 ± 2% relative humidity. Balance precision was better than 5 μg and the gravimetric analysis had an overall limit of quantification (LOQ) = 14 μg in a single weighing.[25] Blank filters were used to assess the quality of the gravimetric analysis but they were not used for correction. The results of the gravimetric analysis were used to calculate the average mass concentration detected by each sampler during the test.

Calibration curve samples and FTIR measurement

Calibration curve samples for DoF FTIR analysis were prepared with two different methods. In the first method, an aliquot of a known concentration (5 mg ml^{-1}) suspension of respirable α-quartz (National Institute of Standard and Technology certified standard reference material, 1878a) in isopropyl alcohol was deposited on a PVC filter.[26] Five different masses range of 5–100 μg of respirable α-quartz (2 sets of samples, total of 10 samples) were prepared and deposit diameter was close to the particle deposit diameter (9-mm) of the EoS cyclone. In the second method, high-purity silica dust (U.S. silica) samples were collected with the EoS cyclones in the calm air chamber (B). Thirteen samples were collected with masses between 9 and 330 μg assessed by gravimetric analysis.

Each calibration sample was then analyzed with a portable FTIR (Alpha, Bruker, Billerica, MA, USA). Transmission IR spectra of the dust-laden filters were acquired. Spectra were collected in transmission mode at 2 cm^{-1} resolution by averaging 40 scans. Interferograms were multiplied by the Blackman-Harris 3-term apodization function prior to Fourier transformation. Spectra were saved from 399.5 to 3998.5 cm^{-1}.

Background correction was applied with a blank filter acquired prior to analyzing a batch of dust samples. This procedure eliminated most of the absorption spectrum of the filter. The α-quartz doublet in the range 816–767 cm^{-1} was integrated because the integrated band area is generally used a calibration metric for quartz quantification.

RESULTS AND DISCUSSION

Size-selective sampling of EoS cyclone

The sampling efficiency curves for EoS cyclones tested with polydisperse glass sphere particles at flow rates of 1.1 and 1.2 l min^{-1} together with the ACGIH/CEN/ISO respirable convention are shown in Figure 3. Each data point and error bars represent the average and standard deviation of five EoS cyclones. The measured average cut off diameters (d_{50}s) for the EoS cyclones at flow rates of 1.1 and 1.2 l min^{-1} were 4.4±0.06 and 4.1±0.15 μm, respectively. The d_{50}s were estimated from curves fitted (using sigmoid, 3 parameters curve fit) to the measured sampling efficiencies of the EoS cyclones. Bias maps of the EoS cyclone were generated from the measured sampling efficiency compared to the ACGIH/CEN/ISO respirable convention and are shown in Figure 4. The bias at the flow rate of 1.1 l min^{-1} ranged from 10 to -4% and those at the flow rate of 1.2 l min^{-1} ranged from 4 to -20 % in particle size distribution of interest when assessing the performance of aerosol sampler (unshaded area in Figure 4).[27] Negative and positive bias indicate an underestimation and overestimation of mass concentration by the EoS cyclone compared to the respirable convention curve, respectively. Based on findings of the d_{50}s and calculated bias between tested flow rates, the flow rate of 1.2 l min^{-1} was selected. A comparison of particle deposition area between 10-mm nylon and EoS cyclone was made by collection of coal dust on PVC filters (Figure 5). The particle deposition diameters in samples collected with the EoS cyclone were measured with samples of coal and gold mine dusts. The average particle deposition diameter of the samples collected with the EoS cyclone was 8.8±0.1 mm in the 60 samples. Diameters of the samples with high-purity silica dust were not measured because it is difficult to define the edge of particle deposition visually due to the similar colors of the high-purity silica dust and the PVC filter.

Respirable dust mass concentration comparison

A total of 180 samples were collected with three different dusts in the calm air chamber (B) and one sample was lost while handling. Dust mass ranges collected with 10-mm nylon cyclones and EoS cyclones were 0.7–1.6 mg and 0.5–1.3 mg, respectively. The average relative standard deviation of the respirable dust mass concentration obtained from the five 10-mm nylon cyclones and five EoS cyclones were < 2% and < 3%, respectively. This indicates not only a low spatial variability inside the chamber (B) but also low sampling variation within both samplers. Average and standard deviation of 5 respirable dust mass concentrations from both 10-mm nylon and EoS cyclones were compared (ratio of average respirable dust mass concentration of EoS cyclone to that of 10-mm nylon cyclone). The average ratios were 1.04±0.02, 1.07±0.02, and 1.04±0.02 for the tests with coal dust, high-purity silica, and gold mine dust, respectively and average ratios of all dusts were 1.05±0.02. Student t-test of log-transformed respirable dust mass concentrations between 10-mm nylon and EoS cyclones showed significant difference ($p<0.05$) in each dust and all dusts results due to small standard deviation. Within each dust, no difference was found between low mass loading and high mass loading (up to 1.3 mg for the EoS cyclone) indicating that the use of the EoS cyclone may not present an issue even for relatively high mass loading. The maximum loading studied here, 1.3 mg, from a full shift sample represents an air concentration of 2.3 mg m^{-3}. Current methods of determining silica in air samples do not perform well when silica is < 1% of sampled dust. The OSHA action level of 0.025 mg m^{-3} respirable crystalline silica implies dust levels >2.5 mg m^{-3} should be avoided in order to be able to demonstrate compliance, because of this 1% limitation.

FTIR measurement in calibration curve samples

The calibration curve samples prepared by three different methods were compared: (1) deposition of a known concentration suspension of respirable α-quartz in isopropyl alcohol, (2) air sampling of high-purity silica dust collected with the EoS cyclones in the calm air chamber (B), and (3) air sampling of high-purity silica dust collected with the 10-mm nylon cyclones in the calm air chamber (B) (previously reported[7]) (Figure 6). A direct quantification model approach was used for the estimation of quartz with the field-portable FTIR.[7] Each data point in the calibration curve was characterized by a FTIR raw entry data (integration value of a characteristic band area in the IR spectrum) and the amount of quartz for that sample. The calibration curve slopes in samples from methods (1) and (2) were similar (0.05, 0.052). These slopes were more than 10 times than the one for the 10-mm nylon cyclone. This means that for the same amount of quartz on the filters, the FTIR raw entry data is ten times higher and this implies a higher measurement sensitivity when the samples are collected with the EoS cyclone. This sensitivity is also evident when the measurement for each sample is divided by the absorbance peak area of quartz relative to the LOQ measurement of the portable FTIR.[1] The FTIR response is more than 5 times higher than the LOQ in low mass loading samples (<10 μg) with EoS cyclones. A similar difference is achieved for samples collected by 10-mm nylon cyclones only at mass levels close to 100 μg. When sampling the same concentration for the same period of time, the EoS cyclone would have 10.5 times larger mass per unit filter area assuming a uniform particle deposition across the filters. If both samplers were run so as to collect the same quantity of quartz, the EoS cyclone would have 14.9 times larger mass per unit filter area, again assuming a uniform particle deposition.

The combination of EoS cyclone and portable FTIR has been tested for the measurement of quartz in air samples. It may be applicable also to the measurement of cristobalite in air samples, but this has not been tested. Therefore, results can be compared to the silica permissible exposure limit (PEL) only when it is known that cristobalite is absent, which is typically the case in many environments. However, since the technique is non-destructive, the absence of cristobalite can be confirmed by later laboratory analysis of the filter. The samples can always be sent for off-site analysis in a laboratory that uses such quality assurance materials and which is accredited for the analysis of silica for confirmation of the result.

Further testing will be necessary to compare quartz measurements using the portable FTIR when a sample was collected the EoS cyclone to standard sampling and analysis procedures, especially in field applications.

Design and calibration of the EOS cyclone

Restricting particle deposition on the sampling filter to an area approximately congruent with the portable FTIR beam may have undesirable consequences. For example, the smaller filter aperture increases the pressure drop across the filter, which increases the strain on the pump and shortens its battery life. However, by reducing the flow to 1.2 l min^{-1}, when using a clean 25-mm polyvinylchloride (PVC, 5 μm pore size) filter, the pressure drop was measured to be only 2.8 times that of the same filter mounted in a standard 25 mm closed-face cassette.

Compared to respirable size-selective samplers fabricated by injection molding with plastic or machining with metals, 3D printing does not produce a smooth surface, especially inside of the cyclone body, which may affect performance. However, based on the findings of the present study, the printing layer lines inside of the cyclone body from the 3D printing may have a minimal effect on size-selective sampling. More advanced 3D printers are available that can print objects with smoother surfaces and smaller tolerances. Advanced materials are likewise available, as are additives to render objects electrostatically dissipative, which would minimize electrostatic issues in air sampling. One of the advantages of the EoS cyclone is its user-friendliness. It is not necessary to remove the filter from its holder for FTIR analysis in field sampling. The filter holder is removed from the cyclone and put in an IR sample holder for the analysis. Removing filters from the filter holder or cassette in field sampling would be cumbersome and could lead to sample loss. Ease of calibration also would be desirable for field sampling but the EoS cyclone requires a calibration jar similar to the 10-mm nylon cyclone. Alternatively, NIOSH and OSHA provide a jarless procedure for cyclone calibration by using different pore size of filters i.e. light load (5 μm pore size; 2–5 inches of water pressure) and increased load (0.8 μm pore size filters in series; 25–35 inches of water pressure).[6, 28]

CONCLUSION

Based on the testing results, the newly developed EoS cyclone showed minimum bias compared to the respirable convention at a flow rate of 1.2 l min^{-1}. The EoS cyclone showed near a 1:1 relationship to the 10-mm nylon cyclone in respirable dust mass concentration (5% larger) and its decreased particle deposition area showed approximately 10 times higher quartz IR absorbance compared to the 10-mm nylon cyclone. Higher IR absorbance may provide better quantification of quartz in low mass loading samples. A combination of the EoS cyclone with the portable FTIR spectroscopy may provide a better strategy for exposure assessment with faster feedback.

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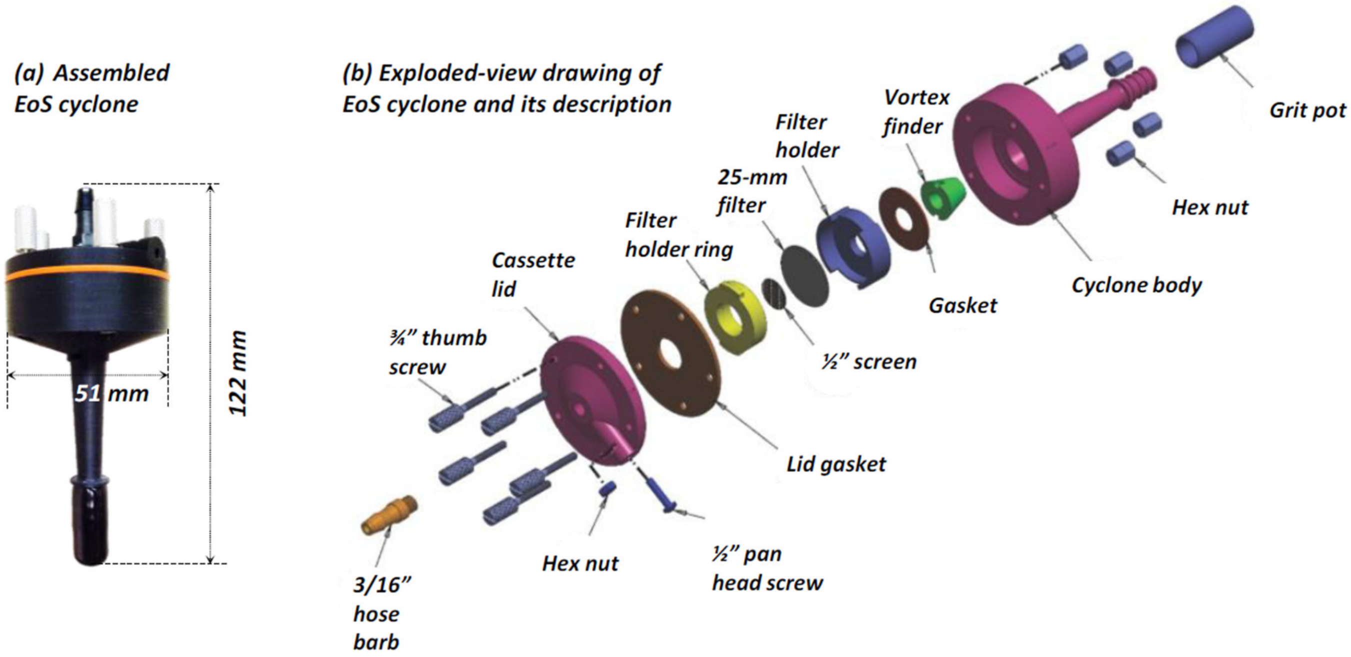


FIGURE 1.
(a) Assembled EoS cyclone and (b) exploded-view drawing of the EoS cyclone.

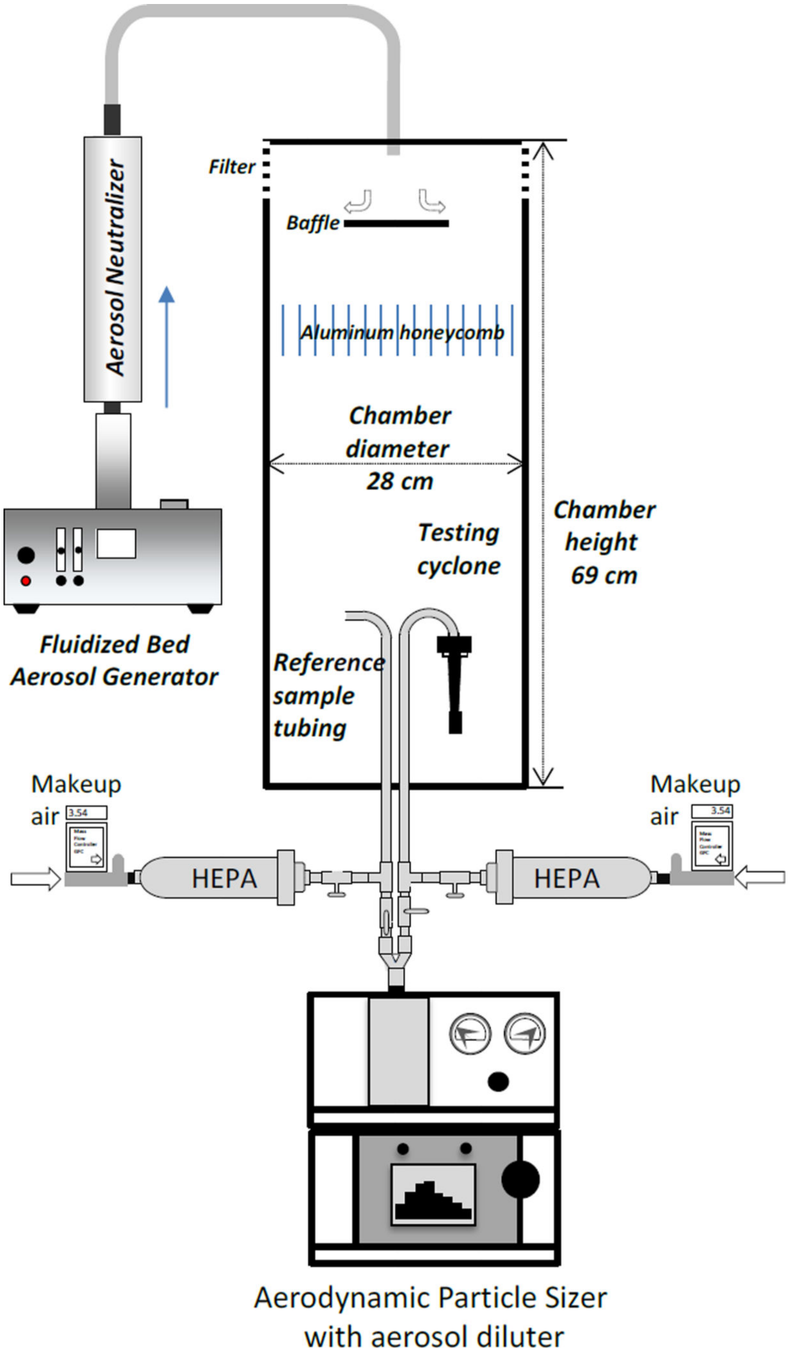


FIGURE 2.
Experimental setup to determine sampling efficiency of EoS cyclone

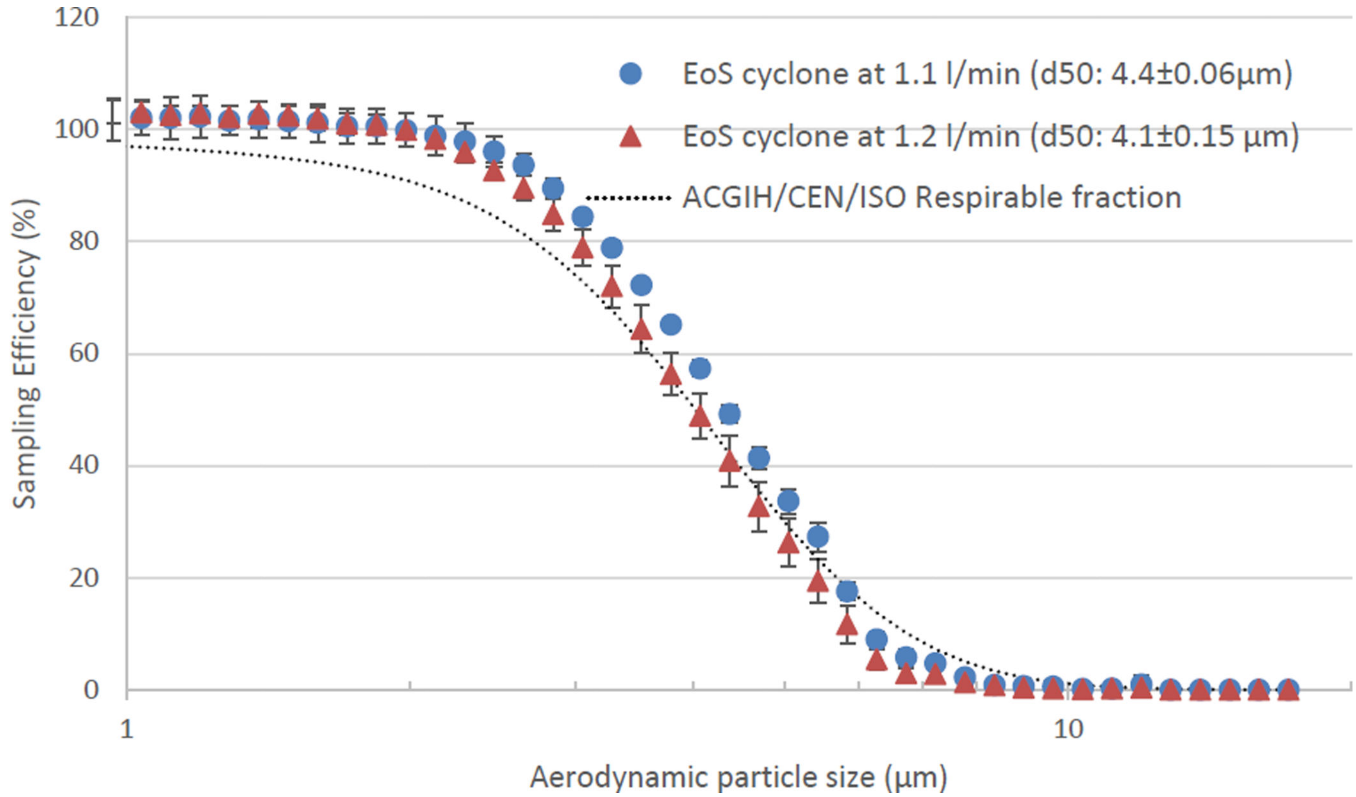
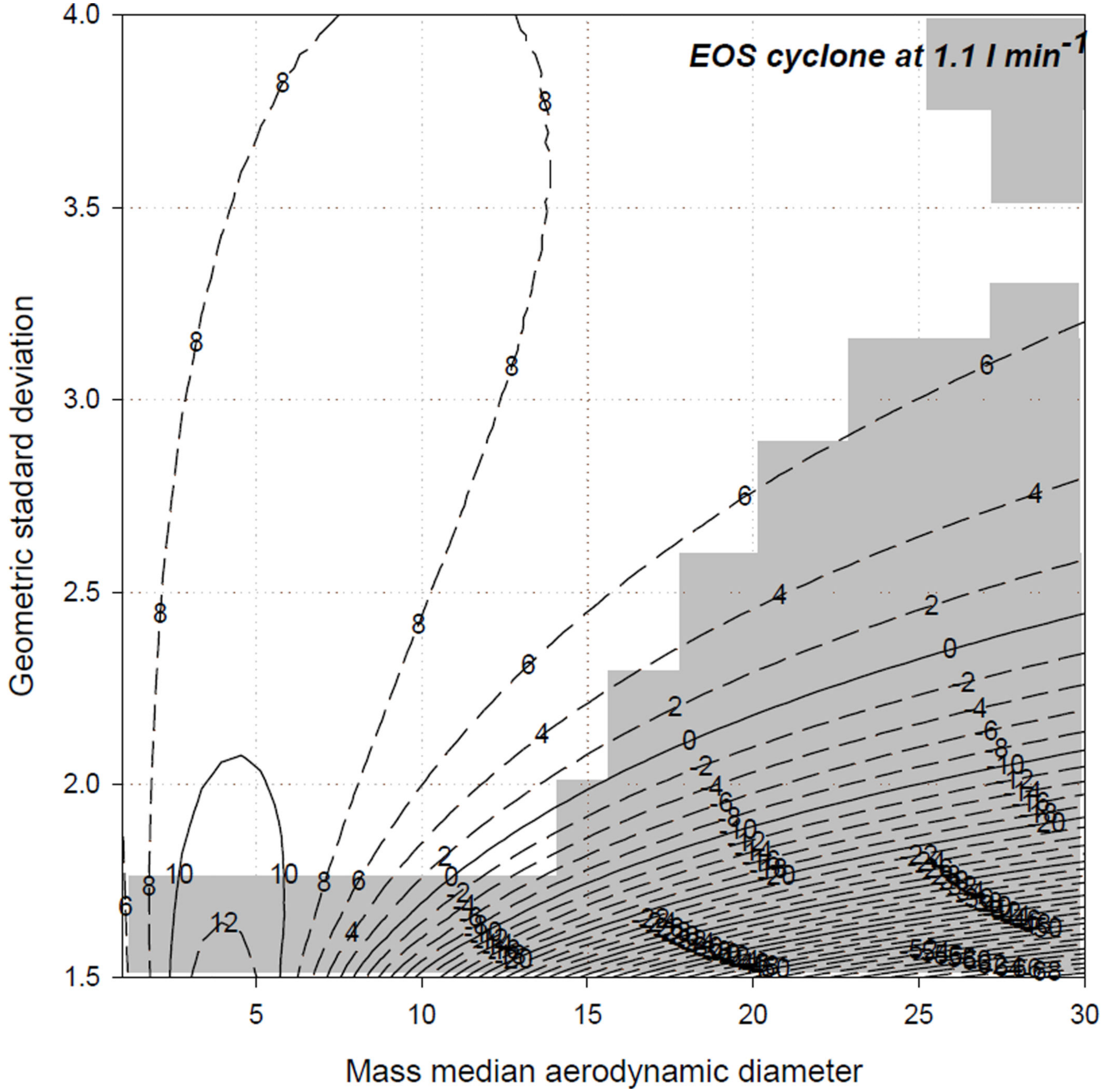


FIGURE 3.

Sampling efficiency of EoS cyclone at the flow rates of 1.1 and 1.2 l min^{-1} together with ACGIH/CEN/ISO respirable convention.


EOS cyclone at 1.1 l min^{-1}
Geometric stadard deviation
Mass median aerodynamic diameter
4.0
3.5
3.0
2.5
2.0
1.5
5
10
15
20
25
30

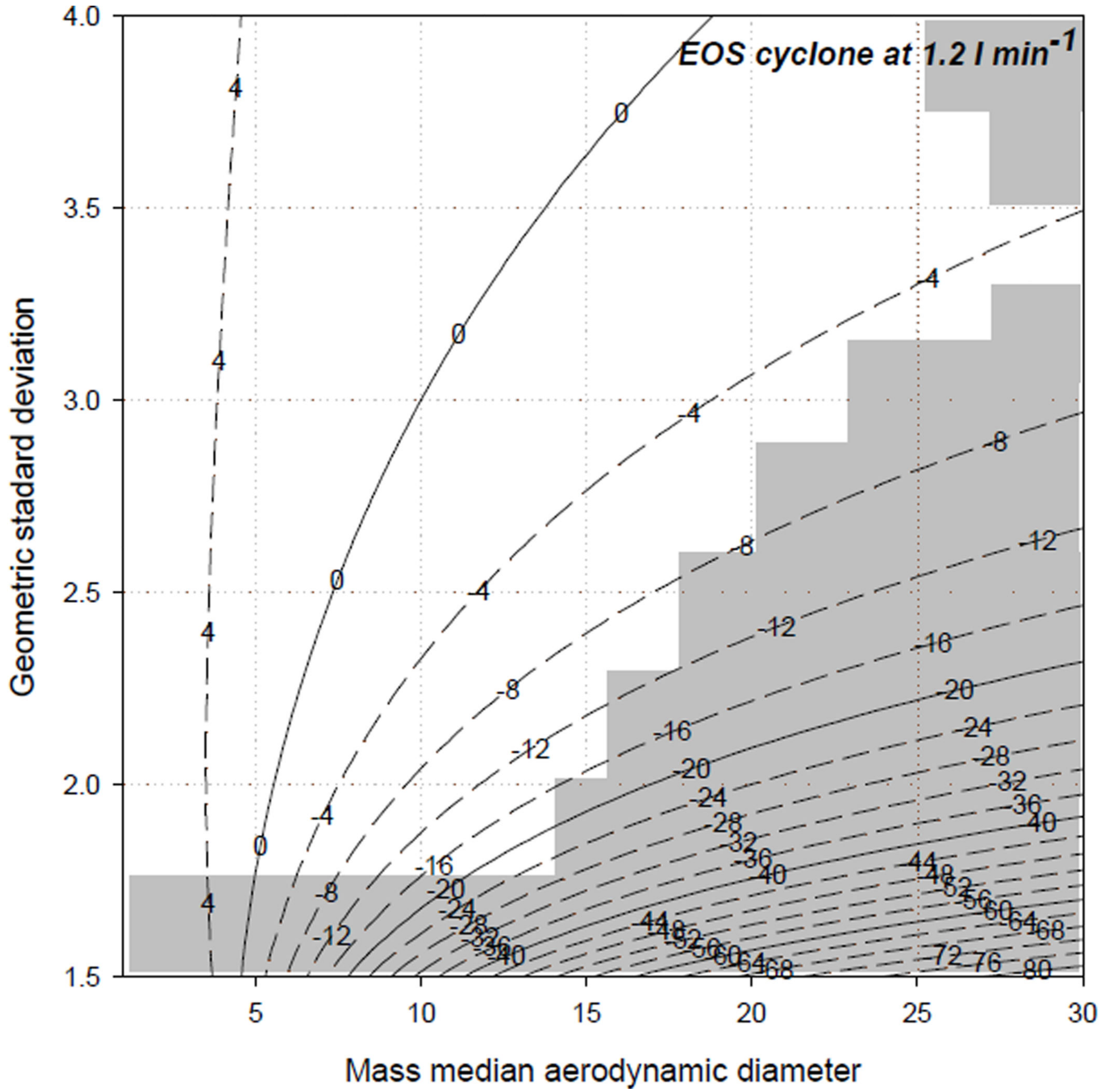


FIGURE 4.
Bias maps of measured EoS cyclone performance at flow rates of 1.1 and 1.2 l min^{-1} compared to ACGIH/CEN/ISO respirable convention. Shaded area represents extreme particle size distributions, which are not included when assessing performance.

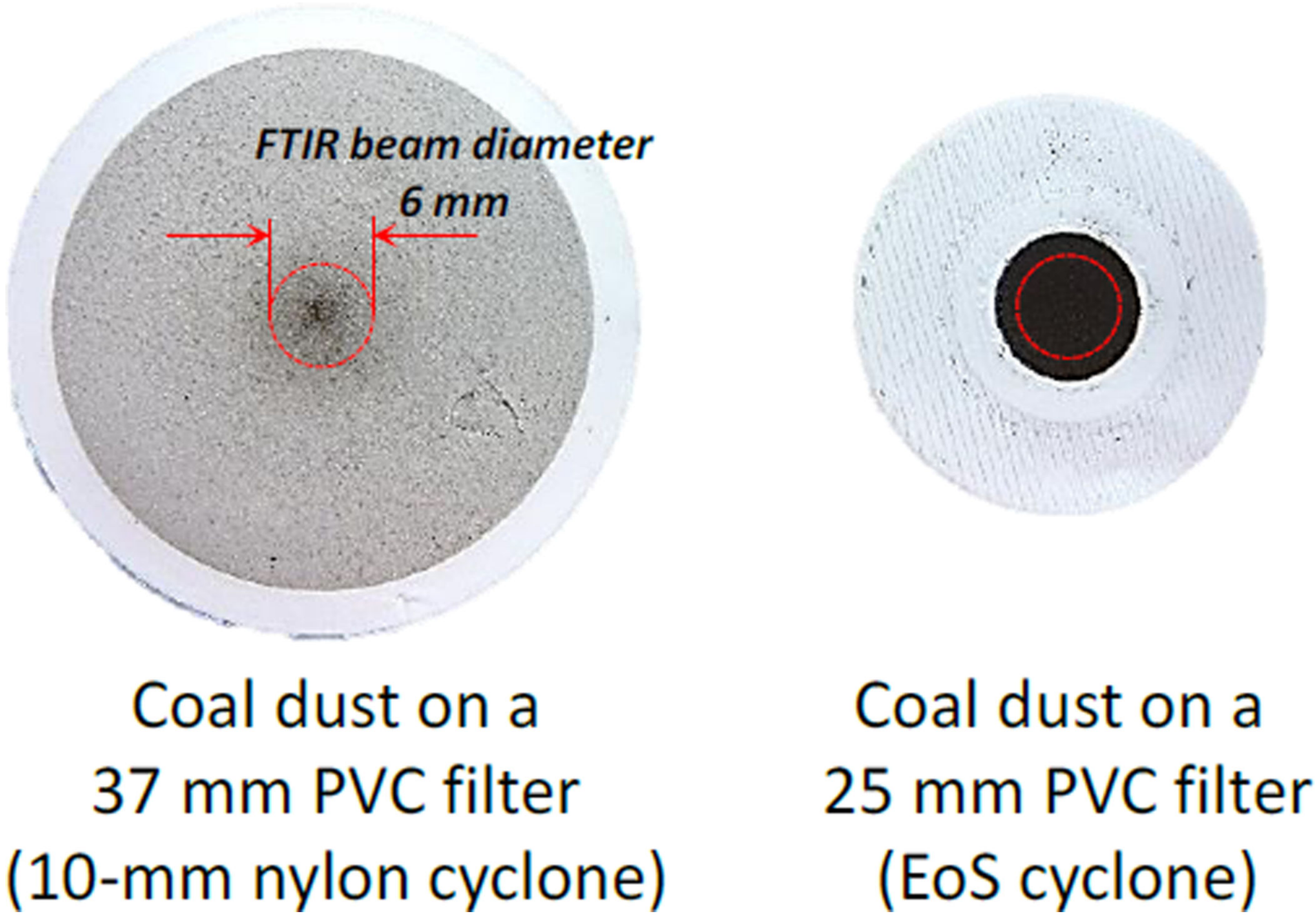


FIGURE 5.

Comparison coal dust deposition area between 10-mm nylon cyclone (37 mm PVC filter) and EoS cyclone (25 mm PVC filter). Dotted line of circles indicate 6 mm beam diameter of portable FTIR.

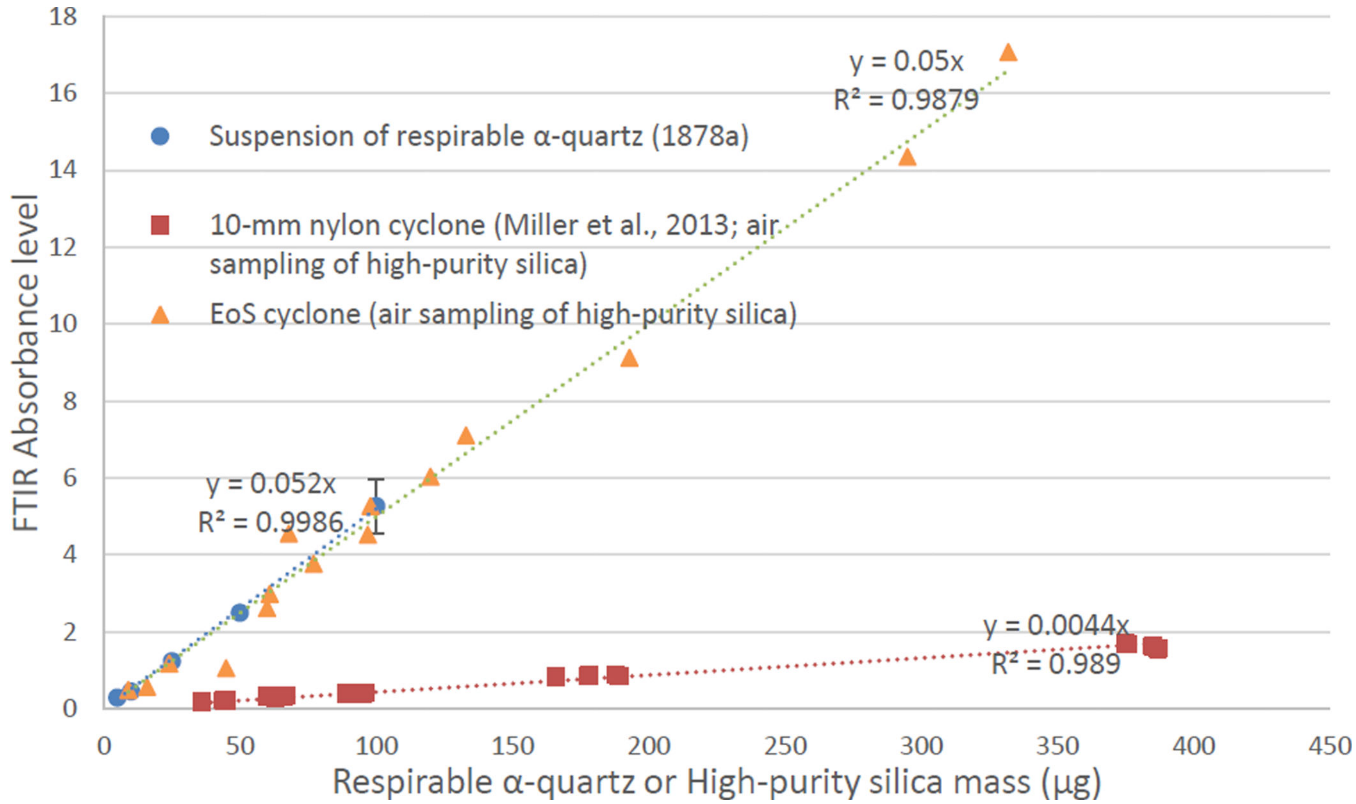


FIGURE 6.

Comparison of calibration curves for the quartz quantification model for samples prepared in three different methods.